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FAB 26

NITROSAMINES

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H. BROOKES
ASSISTANT EDITOR

1

Photolysis of volatile nitrosamines at the picogram level as an aid to confirmation.
Doerr, R. C.; Fiddler, W.
Journal of Chromatography 140 (3) 284-287 (1977) [15 ref. En] [E. Regional Res. Center, USDA, Philadelphia, Pennsylvania 19118, USA]

Solutions containing either the follwing 7 nitrosamines (NA), dimethylnitrosamine, methylethylnitrosamine, diethylnitrosamine, methylbutylnitrosamine, nitrosopiperidine, nitrosopyrrolidine and nitrosomorpholine, or sample extract (fried bacon or other product + 0.5 μg methylethylnitrosamine as an internal standard) are introduced into capillary tubes, sealed and photolysed at 366 nm. Samples are then analysed for NA by GLC-thermal energy analysis, using a 16% Carbowax 20 M-TPA/Gas Chrom P column. For low levels of NA that cannot be confirmed by MS or lack of MS facilities, photolysis at 366 nm of a small portion of sample concentrate offers a simple, rapid and sensitive method for the presumptive evidence of NA; ⩾3 μg NA could be confirmed with a high degree of reliability. SP

2

[What is what in food poisoning. II. Nitrosamines.] [Review]
Goded y Mur, A.
Ion (Madrid) 37 (428) 121-128 (1977) [9 ref. Es]

This review discusses the chemical nature of nitrosamines, their precursors, occurrence in foods, carcinogenic effects and detn. [See FSTA (1977) 9 2C83 for part I.] RM

3

Proceedings of the meat industry research conference, March 25-26, University of Chicago, USA. [Conference proceedings]
United States of America, American Meat Science Association; **United States of America**, American Meat Institute Foundation
Proceedings of the Meat Industry Research Conference 150pp. (1976) [many ref. En]

The full text is given of papers presented at this conference, including: Regulatory aspects and their effect on the meat food industry, by H. C. Mussman (pp. 17-22); Relative risk of nitrate and nitrite ingestion, by S. R. Tannenbaum (pp. 25-33, 37 ref.); Nitrates and nitrites as components of the normal environment, by T. H. Jukes (pp. 41-48, 11 ref.); and Nitrosamine carcinogenesis and latency, by H. B. Jones & A. Grendon (pp. 49-53, 5 ref.). Another 6 papers are abstracted separately, and appear in the author index under **United States of America**, American Meat Science Association [1976 Symposium]. JRR

4

Microwaves cook bacon.
Anon.
Food Engineering International 2 (8) 38-39 (1977) [En, de, fr, es]

2 systems involving microwave heating are described for the pre-cooking of bacon, mainly for the institutional catering and restaurant trade. One system involves cooking with microwaves and the use of hot air to remove moisture. This system is simple and has low capital costs. The other system involves initial heating of the bacon with steam, as microwave generation of heat is inefficient at low temp.; this removes most of the fat and moisture trapped in the fat, hot air then removes more water, and microwave cooking completes the process. The low cooking temp. and short exposure to these temp. are stated to reduce nitrosamine formation in the precooked product; fat stability and colour are better than with conventional pre-cooking processes, and more volatiles are retained, giving better aroma and flavour. The degree of pre-cooking is higher than with conventional pre-cooking, thereby speeding up, and reducing the fat loss in, final cooking. DIH

5

A comparison of proline and putrescine as precursors of N-nitrosopyrrolidine in nitrite-treated pork systems.
Gray, J. I.; Collins, M. E.
Journal of Food Science 42 (4) 1034-1037 (1977) [24 ref. En] [Dep. of Food Sci., Univ. of Guelph, Guelph, Ontario N1G 2WL, Canada]

The role of proline and putrescine as precursors of N-nitrosopyrrolidine was investigated in model and pork systems containing 150 and 1000 p.p.m. sodium nitrite. Both compounds increased the levels of N-nitrosopyrrolidine formed. However, it was concluded from the concn. (30-1000 p.p.m.) of proline and putrescine used in the study that the former was the more likely precursor of N-nitrosopyrrolidine in bacon. The distillate (condensate) collected on heating nitrite-treated pork samples in a heating flask was examined for its N-nitrosopyrrolidine content. Approx. 27-49% of the total N-nitrosopyrrolidine produced was volatilized during the cooking process. IFT

6

Formation of bis(hydroxyalkyl)-N-nitrosamines as products of the nitrosation of spermidine.
Hotchkiss, J. H.; Scanlan, R. A.; Libbey, L. M.
Journal of Agricultural and Food Chemistry 25 (5) 1183-1189 (1977) [16 ref. En] [Dep. of Food Sci. & Tech., Oregon State Univ., Corvallis, Oregon 97331, USA]

The biologically occurring polyamine spermidine was reacted with nitrite in the presence of acid. Reaction products were isolated and converted to trimethylsilyl (TMS) derivatives. The derivatized products were separated by GLC using an all-glass, wall-coated, capillary column. MS data were collected on the chromatographic effluent. 4 isomers of bis(hydroxyalkyl)-N-nitrosamine were synthesized and characterized by IR, NMR and mass spectroscopy and TLC. Kovat's indices were determined for the TMS derivative of each authentic compound and compared to the retention

data collected on the TMS derivatives of the reaction products of spermidine and nitrite. A comparison of the mass spectra of the TMS derivatives of the authentic compounds and reaction products having the same retention time allowed structural assignments to 4 compounds. These were in order of decreasing amounts: 4-hydroxybutyl-(3-hydroxypropyl)-N-nitrosamine, 3-hydroxybutyl-(3-hydroxypropyl)-N-nitrosamine, 4-hydroxybutyl-(2-hydroxypropyl)-N-nitrosamine, and 3-hydroxybutyl-(2-hydroxypropyl)-N-nitrosamine. AS

7

[The nitrate and nitrosamine problem with foods of plant origin.] Das Nitrat-Nitrosamin-Problem bei pflanzlichen Nahrungsmitteln. [Review]
Schwerdtfeger, E.
Qualitas Plantarum - Plant Foods for Human Nutrition 27 (3/4) 339-348 (1977) [many ref. De, en] [Bundesforschungsanstalt für Ernährung, Aussenstelle D-6222 Geisenheim/Rheingau, Federal Republic of Germany]

Aspects considered in this review include: analytical methods for nitrosamines; nitrate and nitrite contents of foods of vegetable origin; effects of var., fertilizers, etc. on nitrate and nitrite concn.; formation of nitrosamines by reaction of nitrites with amines; microbial synthesis of nitrosamines; and inhibitory effects of ascorbic acid on nitrosamine formation. AJDW

8

[Determination of trace N-nitrosopyrrolidine in meat products.]
Kubacki, S.; Danielewska, B.
Prace Instytutow i Laboratoriow Badawczych Przemyslu Spozywczego 25 (3) 409-423 (1975) [12 ref. Pl, ru, en] [Inst. Przemyslu Fermentacyjnego, Warsaw, Poland]

The method is a modified Telling method of increased sensitivity, (sensitivity being 1 μg/kg at 56% yield and 22% coef. of variation). The method consists in steam distillation of the N-nitrosopyrrolidine from the sample in a natural medium. Extraction with dichloromethane and, subsequently, with aqueous KOH solution, was used to purify the distillate after preliminary acidification. The conc. extract is then cleaned with Al_2O_3/MgO. The eluate is analysed by GLC with an electron capture detector. The main deviations from the Telling method are indicated. STI

9

[Nitrite, Clostridium botulinum and nitrosamines.]
Zinck, O.
Dansk Veterinaertidsskrift 60 (8) 347-350 (1977) [Da] [Karensgade 6, Copenhagen Vb, Denmark]

The use of nitrite in meat products is discussed, largely on the basis of a study tour in the USA. Aspects considered include: use of nitrite for control of Clostridium botulinum (with reference to the distribution of Cl. botulinum, frequency of its spores in foods, incidence of botulism and infant botulism, and detection of Cl. botulinum and its toxin); permitted concn. of nitrite in meat products in the USA; distribution of nitrite in cured meats; effects of nitrites on the organoleptic properties of meat products; the possibility of formation of carcinogenic nitrosamines; effects of ascorbic acid on nitrosamine formation; and analytical methods for detn. of nitrosamines. AJDW

10

Control of nitrosamine formation in nitrite cured meat by use of aromatic primary amines.
Bharucha, K. R.; Rubin, L. J.; Cross, C. K. (Canada Packers Ltd.)
United States Patent 4 039 690 (1977) [En]

The formation of nitrosamines in cooked, cured meat products is reduced or eliminated by using aromatic primary amines, e.g. p-alkoxyaminobenzenes, in the curing mixture or to treat the cured meat, e.g. bacon. GL

11

The preferential formation of volatile N-nitrosamines in the fat of fried bacon.
Mottram, D. S.; Patterson, R. L. S.; Edwards, R. A.; Gough, T. A.
Journal of the Science of Food and Agriculture 28 (11) 1025-1029 (1977) [13 ref. En] [Meat Res. Inst., Langford, Bristol, UK]

Whole bacon rashers and the separated lean and fat components were fried in an electrically heated pan and the residues in the pan and the trapped cooking vapours were analysed for N-nitrosopyrrolidine and N-nitrosodimethylamine. The fat produced 12× more N-nitrosopyrrolidine (27.0-42.8 μg/kg wet wt.) and 6× more N-nitrosodimethylamine (12.1-18.8 μg/kg) than the lean (2.0-4.9 and 2.2-3.0 μg/kg, resp.). In all cases the largest proportions (71.5-100%) of both nitrosamines were recovered from the cooking vapours. Prolonged heating of lean did not increase amounts of the nitrosamines. The importance of the non-polar lipid phase of the adipose tissue in nitrosamine formation was demonstrated by heating fat and lean components of bacon in a non-polar heating medium when high levels of nitrosamines were produced from the lean as well as the fat. AS

12

Simple chemiluminescent detector for the screening of foodstuffs for the presence of volatile nitrosamines.
Gough, T. A.; Webb, K. S.; Eaton, R. F.
Journal of Chromatography 137 (2) 293-303 (1977) [24 ref. En] [Lab. of Gov. Chemist, London SE1 9NQ, UK]

The construction and subsequent evaluation of an apparatus for detection of trace amounts of nitrosamines are described. The apparatus consists

of a gas chromatograph, a catalytic chamber to generate nitric oxide from eluted nitrosamines, and a chemiluminescent detector to measure the IR emission resulting from the interaction of this gas with ozone. Examples of the use of the system for determining the nitrosamine concn. in food extracts (luncheon meat, fried bacon, fish, cheese, vegetables) and other materials are given. AS

13

Quantitative aspects of human exposure to nitrosamines. [Review]
Archer, M. C.; Wishnok, J. S.
Food and Cosmetics Toxicology 15 (3) 233-235 (1977) [20 ref. En] [Dep. of Nutr. & Food Sci., Massachusetts Inst. of Tech., Cambridge, Massachusetts 02139, USA]

A method is proposed for the quantitative evaluation of the relative risk of environmental carcinogens and is used to demonstrate that evaluation of the potential dangers of toxic substances on the basis of concn. levels alone may lead to erroneous conclusions. This concept is used to suggest a method of estimating the likelihood that certain environmental carcinogens may cause significant cancer incidence. Finally, these ideas are applied to the problem of nitrosamines in food, and the conclusion is drawn that those present in the highest concn. may not pose the greatest risk. AS

14

Nitrate, nitrite, dimethylnitrosamine and N-nitrosopyrrolidine in some Chinese food products.
Fong, Y. Y.; Chan, W. C.
Food and Cosmetics Toxicology 15 (2) 143-145 (1977) [16 ref. En] [Dep. of Biochem., Univ. of Hong]

Over 60 samples of commonly consumed Chinese food products, including dried shrimps, shrimp sauce and paste, oyster sauce, fish sauce, Chinese sausages and dried squid, were analysed for the presence of dimethylnitrosamine and N-nitrosopyrrolidine. Low levels of dimethylnitrosamine (1-15 parts/billion) and N-nitrosopyrrolidine (2-37 parts/billion) were detected in such foods, and were confirmed in several samples by GC-MS. No correlation was found between the amounts of residual nitrate or nitrite present and the levels of nitrosamines found in the food. AS

15

Group evaluation of volatile and nonvolatile N-nitroso compounds in foodstuffs. [Lecture]
Dikun, P. P.
IARC Scientific Publications No. 14, 57-62 (1976) [3 ref. En] [N. N. Petrov Res. Inst. of Oncology, Min. of Health of the USSR, Leningrad, USSR]

The sequence of operations for analysis of total volatile and non-volatile nitrosamines by a method involving significant reduction in the number of isolation and purification procedures (in order to minimize possible loss of nitrosamines) is given. The main drawback of the method is its relatively low sensitivity, 0.40-0.45 μmol/kg. Analysis of samples of freshly-frozen cod and the same cod after storage for 2 months showed total nitrosamine contents of 0.44 and 0.82 μmol/kg, resp. Smoking the fish gave the following results (μmol/kg): using relatively cold sawdust smoke from a smoke generator, 0.23 and 1.30; with 'Vakhtol' smoking liquor, 0.56 and 0.90; and with conventional wood-fire smoke, 3.8 and 6.0, resp. Thus smoking method and duration of storage affect nitrosamine content. Another group evaluation method (operational sequence given) allowed separate estimation of total contents of volatile and non-volatile (water-soluble) nitrosamines, and showed them to be present in approx. the same ratio (1:4) in both hot-smoked cod and boiled sausage var. 'Doktorskaya'. The possibility of using an analysis method involving measurement of spectra of spontaneous combinational scattering (SCS), or Raman spectroscopy, for detn. and identification is mentioned. [See FSTA (1978) 10 5C147.] AL

16

Techniques for the screening of foodstuffs for volatile nitrosamines. [Lecture]
Gough, T. A.; Woollam, C. J.
IARC Scientific Publications No. 14, 85-96 (1976) [26 ref. En] [Lab. of the Gov. Chem., London, UK]

Current methods for screening foods for presence of volatile nitrosamines are discussed, and their limitations noted. An alternative approach in which nitrosamines are catalytically decomposed after GLC separation to give nitric oxide is considered. The feasibility of using nitric oxide-selective detectors (Pd/Ag alloy tube, chemiluminescent detector, electron-capture detector, or flame-ionization detector) is assessed, and some preliminary findings are presented. [See FSTA (1978) 10 5C147.] AL

17

Analysis of volatile amines contained in foodstuffs as possible precursors of N-nitroso compounds. [Lecture]
Golovnya, R. V.
IARC Scientific Publications No. 14, 237-245 (1976) [19 ref. En] [Inst. of Organo-Element Compounds, Acad. of Sci. of the USSR, Moscow, USSR]

Relative % of volatile amines were determined in sturgeon and salmon caviare, 4- and 10-month old Rossiiskii cheese, and wheat bread, using a gas chromatographic method of identification. About 30-45 mg/kg of the hydrochloric salts of organic bases were found in some products. Qualitative comparison of the amine composition showed that all the foods contained aliphatic amines, including primary, secondary and tertiary amines. Also a great number of compounds with isoalkyl radicals were found; pyrrolidine and piperidine occurred in

all samples. All these compounds can act as precursors of toxic nitrosamines. Since lactic fermentation bacteria are widely used in the production of various food products, and some are present in the digestive tract, the volatile components produced by Streptococcus lactis were analysed after 6, 24 and 30 h of the logarithmic growth phase; relative % of amines are tabulated. It was shown for the first time that aliphatic amines are produced during lactic fermentation; in particular the concn. of the secondary amine isopropyl-n-butylamine increased markedly, viz. from 15.9% at 6 h to 56.1% at 30 h. [See FSTA (1978) **10** 5C147.] AL

18

Efficient degradation of nitrosamines by photolysis. [Lecture]

Polo, J.; Chow, Y. L.

IARC Scientific Publications No. 14, 473-486 (1976) [19 ref. En] [Dep. of Chem., Simon Fraser Univ., Burnaby, British Columbia, Canada]

A photochemical method is described whereby nitrosamines are degraded into parent amines, N_2 and nitrous oxide. The principles of nitrosamine photochemistry and a prototype methodology are discussed. 2 types of laboratory apparatus were constructed, each with a large reservoir for nitrosamine waste; they could degrade 0.01 and 0.15 mol nitrosamines, resp., in 24 h. [See FSTA (1978) **10** 5C147.] AL

19

[Nitrate and nitrite residues in cheeses.]

Chirkina, T. F.

Gigiena i Sanitariya No. 4, 111-112 (1976) [9 ref. Ru] [Vostochno-Sibirskii Tekh. Inst., Ulan-Ude, USSR]

Various types of cheese made in the Buryat and Altai regions of the USSR (Steppe, Kostroma, Dutch-type, Sovetskii, Lithuanian) were analysed. In all cases saltpetre had been used in their manufacture, and all cheeses contained residual nitrites (average 0.140-0.375 mg%). Nitrates were also determined, in average concn. ranging from 0.615 to 1.120 mg%. The implications of these findings are discussed, e.g. possible formation of carcinogenic nitrosamines during storage of the cheese. Use of saltpetre in cheese manufacture is considered inadvisable, and other methods of preventing cheese blowing are recommended (improving microbiological quality of milk, and use of starters with strains inhibiting gas formation). ADL

20

An investigation of the possible presence or formation of nitrosamines in animal feeds. [Lecture]

Juszkiewicz, T.; Kowalski, B.

IARC Scientific Publications No. 14, 375-383 (1976) [9 ref. En] [Dep. of Pharmacology & Toxicology, Vet. Inst., Pulawy, Poland]

Preliminary results of studies are given in which a total of seven 3-5 yr old milking goats received either (i) single oral doses of aqueous solutions of 80 mg sodium nitrite + 100 mg dimethylamine hydrochloride (DMA) + 200 mg diethylamine hydrochloride (DEA)/kg body wt., (ii) fresh kale (containing 3% potassium nitrate on a DM basis) for 3 days, followed by 100 mg DMA + 200 mg DEA/kg, or (iii) 15 g sugar followed after 3 h by 150 mg sodium nitrite + 300 mg DMA/kg. Nitrosamines were not detected in the milk of (i) and (ii) goats 2, 4 and 6 h after administration of amines. In (iii), a trace of nitrosamine was detected in milk just before death occurred at approx. 2 h. [See FSTA (1978) **10** 5C147.] AL

21

Reduction of nitrosodimethylamine content in Cantonese salt fish. [Lecture]

Fong, Y. Y.; Chan, W. C.

IARC Scientific Publications No. 14, 465-471 (1976) [15 ref. En] [Dep. of Biochem., Univ. of Hong Kong, Hong Kong]

The amounts of nitrosatable amines were determined colorimetrically, and levels of (i) nitrosodimethylamine determined by GLC-MS in 3 marine and 3 fresh-water fish after drastic nitrosation with 0.145M sodium nitrite at pH 3; levels in marine fish were found to be much higher. Levels of (i) were much higher in fish preserved with NaCl + sodium nitrite or nitrate than with crude salt or NaCl alone; levels again were higher in marine fish. Amounts of (i) formed in the marine fish Golden thread (Nempterus virgatus) preserved with crude salt or NaCl, with or without added antimicrobial preservatives, were determined. Benzoic acid was found to be a better preservative than the tetracyclines; some of the latter increased the production of (i). When NaCl + benzoic acid was used, no (i) was detected. [See FSTA (1978) **10** 5C147.] AL

22

[Nitrosamines in meat products containing nitrates and nitrites. I. Matured sausages.]

Panizzi, A.; Luppi, G.; Caserio, G.

Industrie Alimentari 16 (7/8) 105-108 (1977) [25 ref. It] [Lab. Chimici Citterio SPA, Rho, Italy]

Contents of (i) nitrates and (ii) nitrites were determined by the method of Follett & Ratcliff [Journal of the Science of Food and Agriculture (1963) 14 (3) 138] in 111 samples of mature raw salami, 83 of them Italian, and 28 imported. Distribution frequencies of (i) and (ii) contents are tabulated. Mean values for (i) and (ii) in Italian salami were, resp. 21 p.p.m. (range, <25-<60 p.p.m.) and 4 p.p.m. (range, 0-<25 p.p.m.). Corresponding values for imported salami were 50 p.p.m. (range <25-315 p.p.m.) and 6 p.p.m. (range, 0-<25 p.p.m.). It is concluded on the basis of these findings and review of the literature that there is no danger of formation of nitrosamines in Italian salami in view

of legal restrictions on use of nitrates and nitrites in their manufacture. SKK

23

Analysis of N-nitrosoproline in raw bacon. Further evidence that nitrosoproline is not a major precursor of nitrosopyrrolidine.
Hansen, T.; Iwaoka, W.; Green, L.; Tannenbaum, S. R.
Journal of Agricultural and Food Chemistry 25 (6) 1423-1426 (1977) [17 ref. En] [Dep. of Nutr. & Food Sci., Massachusetts Inst. of Tech., Cambridge, Massachusetts 02139, USA]

A procedure is described for the extraction of nitrosoamino acids from raw bacon, based on adsorption to Dowex 2X8-100, elution with 1.0M NaCl at pH 1.0, evaporation to dryness and extraction from salts with dichloromethane. Samples processed in this manner were analysed for nitrosoproline (NPro) content by GLC-thermal energy analysis (GLC-TEA) and by high-performance liquid chromatography with photohydrolysis detection. Content of NPro in raw bacon was 70-80 µg/kg. The content of nitrosopyrrolidine (NPyr) in fried bacon was measured by GLC-TEA, and was 44 µg NPyr/kg. Since NPyr is also present in cooking vapour and cooked-out fat, even decarboxylation of all pre-formed NPro would be insufficient for the quantities of NPyr formed in frying. Reported conversions of NPro to NPyr in model systems are <3%. Free proline is suggested as a precursor of NPyr. DIH

24

Method for the determination of nitrosopyrrolidine in meat and meat products at the 1 µg/kg level by means of gas chromatography-electron capture detection. [Lecture]
Kubacki, S. J.; Borys, A.
IARC Scientific Publications No. 14, 109-116 (1976) [10 ref. En] [Dept. of Instrumental Analysis, Inst. of the Fermentation Ind., Warsaw, Poland]

Modifications to the method of Telling et al. [see IARC Scientific Publications (1974) No. 9, pp. 12-17] allows detn. of nitrosopyrrolidine (NPy) at the 1 µg/kg level with about 56% recovery and a coeff. of variation about 22%. Replacement of steam distillation simplifies the technical aspects and shortens the duration of this stage without decreasing its efficiency. Modifications to the gas chromatographic-electron capture detection procedure reduce NPy retention time to about 8 min. Replacement of the 1% solution of dichloromethane in n-pentane by a 20% solution during rinsing of the alumina column results in a higher degree of purity of the eluate and thus in a higher specificity of the method. Results of analyses of standard NPy solution and 2 meat samples spiked with 1 or 60 µg/kg NPy, and recoveries of NPy from samples of canned meat with or without 50% added fat, are presented. [See FSTA (1978) 10 5C147.] AL

25

Determination of eight volatile nitrosamines in thirty cured meat products with capillary gas chromatography-high-resolution mass spectrometry: the presence of nitrosodiethylamine and the absence of nitrosopyrrolidine. [Lecture]
Groenen, P. J.; Jonk, R. J. G.; Ingen, C. van; Noever de Brauw, M. C. ten
IARC Scientific Publications No. 14, 321-331 (1976) [14 ref. En] [Cent. Inst. for Nutr. & Food Res. TNO, Zeist, Netherlands]

5 samples each of 6 cured meat products, (i) cooked ham, (ii) fermented sausage, (iii) raw bacon, (iv) mildly fried bacon, (v) fried minced meat and (vi) smoked meat, were analysed for presence of 8 volatile nitrosamines, nitrosodimethylamine (NDMA), nitrosomethylethylamine, nitrosodiethylamine (NDEA), nitrosomethyl-n-propylamine, nitrosomethyl-n-butylamine, nitrosodi-n-propylamine, nitrosopyrrolidine (NPy) and nitrosopiperidine. Only NDMA and NDEA were found. NDEA occurred in 2 bacon samples (its concn. increasing during mild frying from 4 and 13 µg/kg to 16 and 43 µg/kg resp.) and in 2 samples of (vi), at 7 and 91 µg/kg resp. 7 samples contained NDMA in amounts of 2-6 µg/kg. No NPy was found in (iv); this may have been due to the mild frying method. [See FSTA (1978) 10 5C147.] AL

26

Nitrosamines in cured meat products. [Lecture]
Sen, N. P.; Iyengar, J. R.; Miles, W. F.; Panalaks, T.
IARC Scientific Publications No. 14, 333-342 (1976) [12 ref. En] [Food Res. Lab., Health Protection Branch, Dep. of Nat. Health & Welfare, Ottawa, Canada]

100 samples of specially selected spiced meat products (e.g. sausages, salami, bologna, wieners, meat loaf, canned luncheon meat) were analysed for nitrate, nitrite and volatile nitrosamines. None of the samples contained high nitrosamine levels but many contained traces, generally in the range 2-50 µg/kg. Some contained as many as 4 nitrosamines, viz. nitrosodimethylamine, nitrosodiethylamine, nitrosopiperidine and nitrosopyrrolidine. A few samples were re-analysed after storage for 2 wk at 4° or -20°C; no significant change in nitrosamine levels was detected. The identity of the nitrosamines was confirmed by GLC-high resolution MS. [See FSTA (1978) 10 5C147.] AS

27

Quantitative and qualitative determination of some volatile nitrosamines in various meat products. [Lecture]
Stephany, R. W.; Freudenthal, J.; Schuller, P. L.
IARC Scientific Publications No. 14, 343-354 (1976) [16 ref. En] [Nat. Inst. of Public Health, Bilthoven, Netherlands]

A dual capillary-column, isothermal, single-ion monitoring high-reduction technique was used for analysis of 40 samples of meat products of 8 types: salami-like sausages, cooked and smoked ham, bacon and bacon-like products (fresh and fried), smoked horse and beef meats, fried minced meat, luncheon meat, and miscellaneous. Recoveries of different nitrosamines were determined in 3 extracts of each sample (spiked with nitrosamine before or after extraction, or unspiked). Mean values (corrected for recovery) and range of the 5 nitrosamines occurring naturally in the 8 meat groups are tabulated. No storage effect on nitrosamine content was noted in luncheon meat samples after 4 yr storage in a freezer. The natural occurrence of 0.2-3.9 μg/kg nitrosodi-n-butylamine (NDBA) in some meat samples was confirmed; thus, the use of NDBA as an added internal standard in the analysis of volatile nitrosamines can no longer be recommended. [See FSTA (1978) 10 5C147.] AL

28

Volatile nitrosamines in fried bacon. [Lecture]
Gough, T. A.; Walters, C. L.
IARC Scientific Publications No. 14, 195-203 (1976) [5 ref. En] [Lab. of the Gov. Chem., London, UK]

The effect of changing the initial levels of nitrite (100 and 200 mg/kg) and nitrate (0, 50, 250 and 500 mg/kg) in the cures for bacon (backs and collars) was determined, as was the effect of combinations of storage temp. and time (5° C for ⩽36 days, 15° C for ⩽21 days, and 25° C for 8 days). The amount of nitrosopyrrolidine (NPy) and nitrosodimethylamine (NDMA) occurring in the fried bacon was determined, and their relative distribution in cooked bacon, its cooked-out fat and in the vapours produced during frying were measured. Results, which are presented in tables, show that NPy concn. in cooked bacon were generally higher in high-nitrite samples than in low-nitrite ones, and tended to decrease with storage time, as did the nitrite levels. There was no apparent correlation between NPy concn. and storage temp. (variations were random), or between NPy concn. and initial nitrate level. Distribution of NPy and NDMA was determined in 18 bacons cured with nitrite levels of 100 and 200 mg/kg and nitrate levels of 0 and 500 mg/kg, stored at 5° C for 1-36 days. Higher contents of NPy and NDMA in cooked bacon and its cooked-out fat were associated with high-nitrite cures, but no correlation was found between nitrosamine concn. and initial nitrate level. There was a higher proportion of NPy and NDMA in cooked-out fat than in cooked bacon, but by far the greatest amounts were lost in the vapour produced during cooking (histograms given). [See FSTA (1978) 10 5C147.] AL

29

Phenolic antioxidants and the inhibition of hepatotoxicity from N-dimethylnitrosamine formed in situ in the rat stomach.
Astill, B. D.; Mulligan, L. T.
Food and Cosmetics Toxicology 15 (3) 167-171 (1977) [21 ref. En] [Health, Safety & Human Factors Lab., Eastman Kodak Co., Rochester, New York, USA]

The effect of the widely used edible stabilizers propyl gallate (PG), butylated hydroxyanisole (BHA), butylated hydroxytoluene (BHT) and tert-butylhydroquinone (TBHQ) on intragastric N-nitrosamine formation was studied. Rats were given by gastric intubation, sodium nitrite (125 mg/kg) and dimethylamine (1000 mg/kg) followed immediately by the test compound in dose of 25, 75 or 225 mg/kg. Ascorbate (200 mg/kg) was used as a positive control. TBHQ gave 60% protection against necrosis and appreciably suppressed enzyme activity increases at 225 mg/kg. BHA and BHT at all dose levels, PG at 25 mg/kg and TBHQ at 25 and 75 mg/kg neither demonstrated protective activity nor induced any lethality. Hence at approx. equimolar levels of nitrite and antioxidant, PG and TBHQ exerted an inhibitory effect on nitrosamine formation, but at lower levels all 4 antioxidants tested had no observable effect on the nitrosamine-forming system. AS

30

[Nitrates, nitrites and nitrosamines in milk and milk products - methods of their determination.]
Nitraty, nitrity i nitrozaminy moloka i molochnykh produktov, metody ikh opredeleniya. [Book]
Samodurov, V. A.; Prigorov, N. I.; Deryugina, V. A.
37pp. (1977) [75 ref. Ru] Moscow, USSR; VNII Maslodel'noi i Syrodel'noi Promyshlennosti

Results of studies on the presence of nitrates, nitrites and nitrosamines in milk products are given with data on their concn. in the different products. Carcinogenic properties of nitrosamines and possible pathways of their entry into the organisms are examined. Examples of the detection of these 3 compounds in cheese during ripening are shown. Modern methods for their detn. are presented. FL

31

A model system for the formation of N-nitrosopyrrolidine in grilled or fried bacon.
Coleman, M. H.
Journal of Food Technology 13 (1) 55-69 (1978) [29 ref. En] [Unilever Res. Lab., Colworth House, Sharnbrook, Bedford, UK]

Observations of nitrosamine formation in grilled bacon were used to develop a model system for the formation of N-nitrosopyrrolidine (NNP). The max. temp. attained in the lean and fat, resp., were 100° C and approx. 200° C during grilling; >99% of NNP formed was in the fat. The pyrrolidine content

of heated and unheated pork fat was measured; heating caused the level to rise from 98.7 to 544.5 parts/billion. Addition of proline (800 p.p.m.) to the fat caused an increase in NNP content after heating at 170°C for 10 min from 0.2 to 2550 parts/billion. Ornithine, hydroxyproline, citrulline, arginine and proline, all possible precursors of pyrrolidine found in pork fat, were heated in tetralin. 100 mg proline yielded 9722 μg pyrrolidine; yields from the other compounds were 2-18 μg. Proline at 800 p.p.m. was heated with nitrite concn. of 32-3200 p.p.m. in methanol in sealed tubes at 170°C for 1 h. NNP yields increased with nitrite concn. from 1.5 to 31.1 p.p.m. Yields of NNP varied from 0.65 to 9.1 p.p.m. when proline concn. was varied from 10 to 1000 p.p.m. with 200 p.p.m. nitrite. The effects of heating time and temp. were also investigated on NNP yields. Addition of water at 1-100% v/v to the proline/nitrite/methanol system caused reduction of yields of NNP from 3 to 0.0 (p.p.m.) with increasing water. The formation of NNP from various precursors, and the products given by heating nitrosoproline were also investigated in the sealed tube system. [Continued in following abstr.] DIH

32

A model system for the formation of N-nitrosopyrrolidine in grilled or fried bacon.
Coleman, M. H.
Journal of Food Technology 13 (1) 55-69 (1978) [29 ref. En] [Unilever Res. Lab., Colworth House, Sharnbrook, Bedford, UK]

[Continued from preceding abstr.] A standard model system of $NaNO_2$ (400 p.p.m.) + proline (800 p.p.m.) in methanol, heated in sealed tubes at 170°C was used to investigate the effects of various antioxidants and other compounds on NNP formation. NH_4CNS and lipid hydroperoxides increased NNP yield; chlorogenic acid and caffeic acid increased NNP yield but gallic acid did not. Addition of ascorbic acid ⩽250 p.p.m. caused increasing NNP yields from 2.7 to 37.5 p.p.m.; higher ascorbic acid concn. caused reduction in NNP yields, but these only reached levels similar to controls with 2000 p.p.m. ascorbate. Ethoxyquin at 0.33% w/v was an effective inhibitor of NNP formation after 15 min at 170°C; formation of NNP in the 1st 15 min at 170°C was not affected. DIH

33

[Methods of determining N-nitrosamines in the environment (air, water, soil, foods).] [Review]
Kostyukovskii, Ya. L.; Melamed, D. B.
Gigiena i Sanitariya No. 1, 63-68 (1978) [51 ref. Ru] [Inst. Pitaniya AMN SSSR, Moscow, USSR]

34

[Methods of determination of carcinogenic N-nitrosamines in foods and feedstuffs.] [Review]
Kowalski, B.
Bromatologia i Chemia Toksykologiczna 10 (4) 326-330 (1977) [44 ref. Pl] [Inst. Weterynarii, Pulawy, Poland]

Aspects covered in this review include extraction and purification of the sample; and qualitative analysis and confirmatory tests (e.g. Coulson electrolytic-conductive detector, heat energy analyzer). HBr

35

Estimate of the volatile nitrosamine content of UK food.
Gough, T. A.; Webb, K. S.; Coleman, R. F.
Nature, UK 272 (5649) 161-163 (1978) [13 ref. En] [Lab. of the Gov. Chem., Stamford Street, London SE1, UK]

A survey of the content of volatile nitrosamines (VN) in foods commonly consumed in the UK was carried out using MS and chemiluminescence, and data are summarized to assess the intake of VN from these foods. Using a standard method of analysis [see FSTA (1976) 8 1C59] samples were analysed for (i) N-nitrosodimethylamine, (ii) N-nitrosodiethylamine, (iii) N-nitrosodi-n-propylamine, (iv) N-nitrosodi-n-butylamine, (v) N-nitrosopiperidine, and (vi) N-nitrosopyrrolidine. Results (/kg) showed that were: all 50 samples of fried bacon contained 1-20 μg (occasionally 200 μg) of (vi), 0.08-0.25 μg of (v), ⩽5 μg of (i), and ⩽0.5 μg of unidentified nitrosamines; other cured meats contained the same nitrosamines but only ⩽1 μg of (vi); of 36 samples of uncured meat (uncooked and cooked) and meat extract cubes, 1 contained 2 μg of (i) and 6 had <0.2 μg of dialkyl nitrosamines; both uncooked and fried fish contained (i) but the only other nitrosamine found was 0.01 μg of (vi) in 1 sample out of 70; 5 of 24 samples of salted, pickled, smoked and canned fish contained (i); all common UK varieties of cheese were examined and only (i) was found, usually at much lower levels than in cured meats, and no nitrosamines were detected in 16 of 76 samples; toasted cheese and cheese with nitrate added during manufacture did not contain more VN than untreated cheese; 22 other dairy products contained no heterocyclic nitrosamines but 3 had ⩽0.1 μg of (i); 6 canned desserts (baby foods) were free of VN above 0.01 μg; canned fruit had <0.1 μg of (i) in 5 of 12 samples and 0.09 of (vi) in only 1 sample; 16 different vegetables contained no VN; [continued in following abstr.] AL

36

Estimate of the volatile nitrosamine content of UK food.
Gough, T. A.; Webb, K. S.; Coleman, R. F.
Nature, UK 272 (5649) 161-163 (1978) [13 ref. En] [Lab. of the Gov. Chem., Stamford Street, London SE1, UK]

[continued from preceding abstr.] 3 of 30 soups contained <0.09 μg/kg of (i), 2 contained (iv) at 0.2 and 0.5 μg, and 1 sample contained (v) and (vi) both at 0.06 μg; and 5 of 13 prepacked complete meals contained 0.01-0.3 μg of dialkyl nitrosamines. Laboratory cooked meals consisting of 36 pastry-based foods, 11 stews and casseroles, and 12 fried meals were also examined. Of 493

samples of foods analysed, cured meats represented the major source of VN, followed by fish and cheese, with all other foods containing an average of ≤ 0.06 µg/kg. Approx. weekly intake is estimated at 1 µg of dialkyl nitrosamines and 3 µg of heterocyclic nitrosamines. AL

37

Nitrite additives - harmful or necessary? [Review]
Ravesi, E. M.
Marine Fisheries Review, National Oceanic and Atmospheric Administration 38 (4) 24-30 (1976) [40 ref. En] [Northeast Utilization Res. Cent., Nat. Marine Fisheries Service, NOAA, Emerson Avenue, PO Box 61, Gloucester, Massachusetts 01930, USA]

The question of whether the continued use of nitrites in the preparation of smoke-cured fishery products and processed meat products is necessary for the production of a product safe from deadly bacterial toxin (responsible for outbreaks of botulism) or whether their presence is responsible for the occurrence and formation of highly potent carcinogens in foods and/or the bodies of consumers is reviewed and discussed. AL

38

Occurrence of nitrate, nitrite, dimethylamine, and dimethylnitrosamine in some fermented Nigerian beverages.
Bassir, O.; Maduagwu, E. N.
Journal of Agricultural and Food Chemistry 26 (1) 200-203 (1978) [31 ref. En] [Biochem. Dep., Univ. of Ibadan, Ibadan, Nigeria]

The contents of NO_3^-, NO_2^- and dimethylamine (DMA) were determined in various fermented Nigerian beverages (palm-wine; burukutu and pito, fermented cereal beverages; ogogoro, a palm-wine or molasses distillate; and nono, skimmed fermented cow's milk). Results are tabulated for beverages purchased from 15 locations in Kwara and Benue States. NO_3^- was found in 80% of the beverages (i.e. in all but the ogogoro samples) in concn. of 6-91 p.p.m.; NO_2^- was present in 60% of the beverages (never in ogogoro) at 0.86-2.9 p.p.m. DMA was present in all beverages at 4.06-31.6 p.p.m. The limits of detection for NO_3^-, NO_2^- and DMA were 1, 0.2 and 2 µg/ml, resp. Dimethyl nitrosamine (DMN) was detected in 1 sample of palm-wine at 0.6 ng/ml. NO_3^-, NO_2^- and DMA were determined colorimetrically and spectrophotometrically, DMN was determined by TLC and GLC and confirmed by MS. DIH

39

Occurrence and determination of N-nitroso-3-hydroxypyrrolidine in cured meat products.
Janzowski, C.; Eisenbrand, G.; Preussmann, R.
Journal of Chromatography 150 (1) 216-220 (1978) [8 ref. En] [Inst. for Toxicology & Chemotherapy, German Cancer Res. Cent., Im Neuenheimer Feld 280, D-6900 Heidelberg, Federal Republic of Germany]

An analytical method developed for the isolation and detn. of (i) N-nitroso-3-hydroxypyrrolidine from foods is described. Clean-up procedure consists of methanol-water (9:1) extraction of the food followed by several liquid-liquid partitions. (i) is determined by gas chromatography (GC) of the concentrate before and after trifluoroacetylation; a thermal energy analyser is used as GC detector. Identity of (i) is confirmed by GC-high resolution MS. N-nitroso-4-hydroxypiperidine is used as an internal standard. Of 33 fried samples analysed for (i), 3 of 10 bacon samples and 1 of 10 boiled ham samples contained 1 part/billion and 5 of 13 Bologna sausage samples contained 1-7 parts/billion. AL

40

[Nitrosamines in cheese?]
Elgersma, R. H. C.
Zuivelzicht 70 (11) 265-267 (1978) [10 ref. Nl, en] [Nederlands Inst. voor Zuivelonderzoek, Ede, Netherlands]

In view of recent reports that cheese imported into Canada contained up to ≤ 20 µg volatile N-nitrosamines/kg, analyses were carried out at 3 laboratories (in Canada, Netherlands and UK) on Gouda and Baby Edam cheeses shortly after manufacture, after storage in the Netherlands or UK, and after export and normal retailing in Canada. The Canadian laboratory, using GLC/MS, detected 1-2 µg N-nitrosodiethylamine/kg in 2 of 14 samples (one freshly made and the other retailed in Canada). This amount corresponded to the detection limit of the method used. The other laboratories, using more sensitive methods (GLC with a thermal energy analyser, detection limit about 0.01 µg/kg), detected only N-nitrosodimethylamine (which was not detected by the Canadian laboratory) in very small amounts, ≤ 0.15 µg/kg. The conditions of storage and export did not appear to affect nitrosamine content, as was confirmed by a further similar study on cheeses purchased at random in Canada. Nitrosamine content was not related to nitrate content of the cheese or to the amount of nitrate used in manufacture. ADL

41

Precursors of dimethylnitrosamine in fried bacon.
Gray, J. I.; Collins, M. E.; MacDonald, B.
Journal of Food Protection 41 (1) 31-35 (1978) [21 ref. En] [Dep. of Food Sci., Univ. of Guelph, Guelph, Ontario N1G 2W1, Canada]

The formation of dimethylnitrosamine (DMN) was studied in model systems and in fried pork samples. The model systems used were (i) a low moisture carboxymethylcellulose system (simulating the dry surface of cured meat products), and (ii) an oil system heated under reflux. Studies of the effect of pH on DMN formation in a (i) system containing 5mM $NaNO_2$ + 1mM precursor (dimethylamine (DMA), trimethylamine, sarcosine or choline

chloride) showed that max. formation of DMN was given at pH 3.4; sarcosine and DMA were the main precursors (3.19 and 2.35% conversion at pH 3.4, after 2 h at 150°C, resp.). The effect of temp. (100-200°C) on the formation of DMN in the (i) system was studied with sarcosine; max. formation was at 140°C (3.23% conversion to DMN at pH 3.4 after 2 h). Higher temp. were thought to volatilize DMN; DMN formation in the (ii) system increased with temp., 5.07% sarcosine in the presence of 5mM $NaNO_2$ was converted to DMN after 15 min at 200°C. 22.29% of N-nitrosarcosine (at 1mM initially) was converted to DMN after 15 min at 200°C in a (ii) system. Phosphoryl choline chloride and soy lecithin were also studied in a (ii) system at 1mM concn. with $NaNO_2$ at 5mM, their respective % conversion to DMN after 30 min at 180°C was 0.99 and 0.43. Formation of DMN from these precursors increased with temp. in the range 120°-180°C. Formation of DMN, and distribution between vapour and meat phases, was studied with fried pork samples containing various amounts of $NaNO_2$ (0-1000 p.p.m.) and added precursors (0, sarcosine at 200 p.p.m. or phosphoryl choline chloride at 1000 p.p.m.). Formation of DMN increased with temp., and $NaNO_2$ and precursor concn. but yields were much lower than in the (ii) system, and ≤80% of the DMN was found in the vapour phase. Highest DMN concn. in the fried material was 135 ng/(g pork before heating) from sarcosine. DIH

42

Formation of N-nitrosopyrrolidine in fried bacon.
Gray, J. I.; Collins, M. E.
Journal of Food Protection 41 (1) 36-39 (1978) [25 ref. En] [Dep. of Food Sci., Univ. of Guelph, Guelph, Ontario N1G 2W1, Canada]

Formation of N-nitrosopyrrolidine (N-Pyr) from proline and N-nitrosoproline (N-Pro) was investigated by cooking pork slices to which these precursors had been added. When the cooking system was heated in an oil bath at 200°C for 12 min, 0.33 and 2.18% yields of the N-nitrosamine were obtained from proline and N-Pro, resp. The N-Pro contents of pork slices to which 2 levels of nitrite were added were determined after 1 and 8 days of storage at 2°C. Results indicate that the formation of N-Pro occurs at such a rate that 1 day after addition of nitrite there is theoretically sufficient N-Pro formed to account for the quantities of N-Pyr isolated from cooked bacon. However, the rate of decarboxylation of the initial N-Pro in raw bacon is not great enough to account for the N-Pyr isolated from cooked bacon. [See also preceding abstr.]. AS

43

The nitrosation of foods.
Newton, B. E.
Dissertation Abstracts International, C 37 (3) 603 (1977) [En] [Univ. of Surrey, Guildford, Surrey, UK]

A quantitative measure of the amounts of nitrosatable compounds present in various foods was obtained by reacting whole foods with a large excess of $NaNO_2$ and studying the amount of N-nitroso compounds formed. This treatment produced several volatile N-nitrosamines, identified by combined gas chromatography-MS, although the major products were non-volatile nitrosamines of unknown identity. The same foods were exposed to low concn. of $NaNO_2$ (10 and 100 p.p.m.) in a medium simulating the human stomach to measure the probable nitrosation that may occur in the human stomach, after a nitrite-containing meal is consumed. Nitrosation of foods did occur, the products being mainly non-volatile N-nitroso compounds. In vivo studies were performed, where nitrite-containing meals were fed to humans. No nitrosation could be detected and it was observed that the ingested nitrite disappeared rapidly from the stomach contents. Catalysis of nitrosations by various biological anions was examined, but none produced a marked catalytic effect and thiocyanate remained the most effective catalyst. Concn. of thiocyanate, comparable to levels found in human saliva, were incorporated into incubations of food with nitrite in the simulated stomach medium, but there was no great enhancement in the amount of nitrosation that occurred. The possible nitrosation of peptide bonds was studied, using glycyl glycine. It was demonstrated that an N-nitroso derivative could also be nitrosated, but the nature of the constituent amino acids did not markedly affect the nitrosation. Several amines present in foods, were examined for their potential for nitrosation. AS

44

[Nitrosamines in meat products containing nitrates and nitrites. II. Italian mortadella.]
Caserio, G.; Luppi, G.; Panizzi, A.
Industrie Alimentari 17 (1) 45-46 (1978) [17 ref. It] [Cattedra di Patologia Anim. e Ispezione delle Carni, Univ. di Milano, Milan, Italy]

Following previous work on the nitrite and nitrate contents of Italian salami [see FSTA (1978) 10 5S663] the survey was extended to Italian mortadella products to assess the possible risk of nitrosamine formation (assumed to depend on the quantity of residual nitrite and nitrate) in view of the cooking temp. of 75°C at the centre. Nitrate and nitrite contents were determined in 90 samples of mortadella taken over 4 months, representing normal products manufactured with 150 p.p.m. $NaNO_2$ but with no addition of nitrate. Results are tabulated, with distrobution in ranges between nil and 30 p.p.m. nitrite and 50 p.p.m. nitrate. Average of all values was 9.5 p.p.m. nitrite and 24.14 p.p.m. nitrate. All samples were well below the FAO/WHO recommended max. and the US limit (for cooked products) of 100 p.p.m., and satisfy the legal requirements of various other countries. ELC

45

[Determination of the N-nitrosamine content of meat and some meat products.]
Pokrovskii, A. A.; Kostyukovskii, Y. L.; Melamed, D. B.; Medvedev, F. A.
Voprosy Pitaniya No. 2, 65-72 (1978) [17 ref. Ru, en] [Inst. Pitaniya AMN SSSR, Moscow, USSR]

The contents of carcinogenic nitrosamines (dimethylnitrosamine (DMNA), diethylnitrosamine (DENA) and nitrosopiperidine (NP)) were determined in 9 types of sausage, 1 of meat loaf, 1 of ham, 1 each of fresh and stored (3 months at -10° to -12°C) beef and 1 of sausage meat by fluorimetry, chromatography/MS and MS (the latter 2 methods being used to confirm the presence after a positive fluorimetry result). Numbers of samples ranged from 7 to 22 for each product. Mean ranges found (μg/kg) were 0.5-5.4, 1.0-6.1 and 0.9-2.4 for DMNA, DENA and NP, resp. (with, additionally, a mean of 23.4 for NP in 12 samples of 1 type of sausage). HBr

46

[Nitrosamine formation in ripened meat products.]
Zur Nitrosaminbildung in gereiften Fleischwaren.
Kotter, L.; Schmidt, H.; Fishcer, A.
Archiv für Lebensmittelhygiene 28 (1) 8-11 (1977) [35 ref. De, en] [Inst. für Hygiene & Tech. der Lebensmittel Tierischen Ursprungs, Univ. München, Veterinärstrasse 13, 8000 Munich 22, Federal Republic of Germany]

See FSTA (1977) 9 12S2137.

47

Occurrence of methylguanidine and agmatine, nitrosatable guanidino compounds, in foods.
Kawabata, T.; Ohshima, H.; Ino, M.
Journal of Agricultural and Food Chemistry 26 (2) 334-338 (1978) [16 ref. En] [Dep. of Biomed. Res. on Food, Nat. Inst. of Health, Sinagawa-ku, Tokyo 141, Japan]

Among naturally occurring guanidines, nitrosated methylguanidine (MG) has been reported to be strongly mutagenic and carcinogenic and nitrosated agmatine (AG) to be moderately mutagenic. A survey was conducted on the MG and AG contents of various fresh and processed foods. No appreciable amount or trace amounts of MG could be detected in fresh beef, pork, chicken, and various fish and shellfish; in addition, almost the same low levels of MG were detected in various processed fish and meat foods, except for the Japanese smoked-dried fish products Katsuo-bushi and Kezuri-bushi; MG values for these products ranged from 18 to 178 mg/kg. Comparatively high concn. of AG were detected in fresh abalone and top-shell muscles ranging from 40 to 200 mg/kg among the fresh foods tested, while fairly high concn. of AG could be detected in some processed foods; dried squid was found to contain as much as 650 mg AG/kg. AS

48

The presence of dimethyl- and diethyl-nitrosamines in deionized water.
Fiddler, W.; Pensabene, J. W.; Doerr, R. C.; Dooley, C. J.
Food and Cosmetics Toxicology 15 (5) 441-443 (1977) [8 ref. En] [E. Regional Res. Cent., Philadelphia, Pennsylvania 19118, USA]

The application of the GLC-Thermal Energy Analyser (TEA) to the detection of apparent nitrosamines in deionized water is described. 27 of 42 samples of water exposed to deionizing resins contained 0.03-0.34 p.p.b. (μg/l.) dimethylnitrosamine (DMNA). 12 of these 27, mainly from water obtained after resin regeneration, were confirmed as containing DMNA by GLC and high resolution MS. 2 of the samples of deionized water contained 0.33 and 0.83 μg diethylnitrosamine/l. as well as the DMNA. The origin of the nitrosamines is not known at present. VJG

49

Volatile nitrosamines from ion-exchange resins.
Gough, T. A.; Webb, K. S.; McPhail, M. F.
Food and Cosmetics Toxicology 15 (5) 437-440 (1977) [10 ref. En] [Lab. of the Gov. Chemist, Stamford Street, London SE1 9NQ, UK]

Trace amounts of dimethylnitrosamine (DMNA), but no other nitrosamines, were detected in some samples of the deionized water which was prepared from tap water. Ion-exchange resins, including those used in water deionization plants, were examined and the anion exchangers were identified as the source of the nitrosamines. Gas chromatography combined with chemiluminescence and MS was used to identify and quantify the nitrosamines. The presence of nitrosamines in deionized water may result in contamination of samples which are being analysed for nitrosamines. On the basis of the amounts of DMNA found in the deionized water samples, the contribution toward the dialkylnitrosamine content of a food extract, and hence the apparent nitrosamine level in the original food, can be calculated. VJG

50

[Hygienic and chemical aspects of N-nitroso compounds.] [Review]
Tanimura, A.
Journal of Hygienic Chemistry [Eisei Kagaku] 22 (5) 245-253 (1976) [41 ref. Ja, en] [Nat. Inst. Hygiene Sci., Setagaya-ku, Tokyo, Japan]

A review is given of N-nitroso compounds which might be formed secondarily in various foods, with references to past literature on the carcinogenicity and mutagenicity of the N-nitroso compounds. Special references are made to recent literature on N-nitroso compounds detected in daily food, distribution of nitrogenous compounds and nitrites which are considered to be precursors, inhibitive and accelerative factors of N-nitrosation reaction, nitrosation of pharmaceuticals and agricultural

chemicals, carcinogenicity of N-nitroso compounds, and correlation between food and human cancer. AS

51

[Determination of n-nitrosamines in water from their fluorescent derivatives.]
Melamed, D. B.; Kostyukovskii, Ya. L.
Gigiena i Sanitariya No. 5, 67-68 (1978) [3 ref. Ru] [Inst. Pitaniya AMN SSSR, Moscow, USSR]

52

Nitrosamine formation in home-cooked bacon.
Wasserman, A. E.; Pensabene, J. W.; Piotrowski, E. G.
Journal of Food Science 43 (1) 276-277 (1978) [7 ref. En] [E. Regional Res. Cent., USDA, Philadelphia, Pennsylvania 19118, USA]

Randomized slices of bacon were cooked at home by 25 consumers by their usual method of preparation and the edible portions were analysed for nitrosamines. N-nitrosodimethylamine (DMNA) and N-nitrosopyrrolidine (NPyr) were found in all samples. Of the 25 preparations, 5 contained DMNA and 5 contained NPyr in concn. >10 μg/kg; only 1 sample contained >10 μg/kg of both nitrosamines. Although the association between nitrosamine concn., time and amount of heat is not strong, it appears that frying bacon at low or medium heat for <10 min can result in <10 μg/kg DMNA or NPyr. IFT

53

Nitroso compounds and carcinogenesis.
Cooper, P.
Food and Cosmetics Toxicology 16 (1) 69-71 (1978) [20 ref. En] [British Ind. Biol. Res. Ass., Woodmansterne Rd., Carshalton, Surrey, UK]

Consideration is given to: minimization of nitroso compound formation; the role of drugs in nitrosation; the possible hazard of urea derivatives as nitrosation substrates; and amino acids and heterocyclic compounds and carcinogenesis. VJG

54

[Evaluation of nitrate, nitrite and N-nitroso compounds.] Zur Bewertung von Nitrat, Nitrit und N-Nitrosoverbindungen. [Review]
Sieber, R.; Blanc, B.
Deutsche Molkerei-Zeitung 99 (8) 240-242, 244-246, 248; (9) 278-280, 282 (1978) [150 ref. De] [Eidgenössische Forschungsanstalt für Milchwirtschaft, CH-3097 Liebefeld-Bern, Switzerland]

The authors review the literature to date on the significance, from the health hazard point of view, of the presence of nitrate and nitrite in food materials as factors in the formation of carcinogenic N-nitrosamines. Data for nitrate and nitrite contents of various foods, especially dairy products, are presented, and WHO recommended limits for daily intake of each are set out. Conditions favouring formation of N-nitrosamines by interaction of nitrite and secondary amines during preparation and during subsequent digestion of foods are discussed. Attainment of satisfactory min. levels of nitrosamines would appear to depend on elimination of processing methods which favour formation of nitrosamines; and availability, for control purposes, of foods known to be low in nitrate-nitrite and in nitrosamine-forming amines. GTP

55

A sensitive method for the colorimetric determination of volatile nitrosamines in food products and air.
Ceh, L.; Ender, F.
Food and Cosmetics Toxicology 16 (2) 117-121 (1978) [16 ref. En] [Dep. of Biochem., Vet. Coll. of Norway, PO Box 8146, Dep. Oslo 1, Norway]

A quantity of food containing ≥ 0.1 μg nitrosodialkylamine, but preferably 1-10 μg, was extracted for 45 min with a vol. of freshly distilled water equal to 5 × the dry wt., at a temp. of about 90-95°C in a boiling water bath. The heated mass was filtered, washed twice with distilled water and concentrated by distillations from alkaline and acid solution. For smoked fish and smoked meat products a further purification step was needed to remove indole derivatives. The nitrosamines were then reduced with Zn and HCl, followed by oxidation to monoalkylhydrazines and coupling of these with p-dimethylaminobenzaldehyde. Photometry of the resulting alkylaldazine complex at 458 nm, allows determination of N-nitrosodimethylamine in amounts down to 0.1 μg. Factors affecting the yield of the alkylaldazine complex and optimum conditions for the determination are discussed. This method has been used for the determination of N-nitrosodimethylamine in food products at concn. as low as 0.5 μg/kg. VJG

56

[Effect of a dose of nitrate on the salivary and gastric nitrite of man.]
Klein, D.; Gaconnet, N.; Poullain, B.; Debry, G.
Food and Cosmetics Toxicology 16 (2) 111-115 (1978) [18 ref. Fr, en] [Dep. de Nutr. et des Maladies Metaboliques de l'Univ. de Nancy I, Nancy, France]

Following ingestion of a test meal containing 112 mg nitrate, the rate of evolution of salivary and gastric nitrite and nitrate was studied in 2 groups of volunteers. The possible relevance of the findings to nitrosamine formation in vivo is discussed. VJG

57

Detection and determination of N-nitrosamino acids by thin-layer chromatography using fluorescamine.

Young, J. C.

Journal of Chromatography 151 (2) 215-221 (1978) [58 ref. En] [Chem. & Biol. Res. Inst., Agric. Canada, Ottawa, Ontario K1A OC6, Canada]

A novel procedure is described for the detection and detn. of N-nitrosamino acids (NAAs) on activated silica gel TLC plates. N-nitrososarcosine, N-nitrosoproline, and N-nitroso-4-hydroxyproline could be detected as fluorophors at the 200-pmole level (20-30 ng) after being irradiated with UV light and sprayed with fluorescamine reagent. Spectrophotometric detn. of the relative fluorescence of 0.4-40 nmoles of NAAs gave rise to similar calibration curves when plotted on a log-log scale. An application of this method to the detection of NAAs in uncooked bacon is described. AS

58

The thermal energy analysis of sodium saccharin.

Krull, I. S.; Goff, U.; Wolf, M.; Heos, A. M.; Fine, D. H.; Arsenault, G. P.

Food and Cosmetics Toxicology 16 (2) 105-110 (1978) [38 ref. En] [Cancer Res. Div., Thermo Electron Corp., 45 First Avenue, Waltham, Massachusetts 02154, USA]

In view of the possible formation of N-nitroso impurities during the manufacture of sodium saccharin by the Maumee process (the process used by Sherwin-Williams the major US supplier) a study was undertaken to determine the presence of any N-nitroso materials in a commercial sample of sodium saccharin, applying methods and techniques similar to those used in the past to determine N-nitroso compounds. Sodium saccharin has been found to elicit a positive response, equivalent to about 0.0001 mol of an N-nitroso compound in thermal energy analysis (TEA). All efforts to ascribe this response to an N-nitroso impurity have been unsuccessful. It is concluded that the weak TEA response observed is due to the decomposition of sodium saccharin itself. VJG

59

Positive interferant in the chemiluminescent detection of nitrosamines.

Gough, T. A.; Webb, K. S.

Journal of Chromatography 154 (2) 234-237 (1978) [13 ref. En] [Lab. of Gov. Chem., Stamford Street, London SE1 9NQ, UK]

Previous studies of nitrosamine content in foods using gas chromatography with chemiluminescent detection have not given false positive results at nitrosamine retention times, after MS confirmation. Interference with chemiluminescent detection by a compound having a retention time equal to N-nitrosodi-n-propylamine on a Carbowax 20 M GLC column is reported. The compound was present in water extracts of the anion-exchange resin Deacidite FF, and also gave false positive values for N-nitrosodiethylamine on Apiezon L GLC columns with chemiluminescence detection. Tentative MS identification of the compound as N-dimethylbenzylamine was made. It is recommended that MS be used to confirm presence of nitrosamines detected by chemiluminescence, at least with materials not extensively studied. DIH

60

Nitrosamines in feed, milk and milk products.

Terplan, G.; Kalbfuss, W.; Hallermayer, E.; Unsinn, P.; Heerdegen, C.

XX International Dairy Congress E, 37-38 (1978) [4 ref. En] [Lehrstuhl für Hygiene & Tech. der Milch, Univ. München, Munich, Federal Republic of Germany]

Diethyl-, dipropyl- and diisopropyl-nitrosamine (NA) were detected at 2-33 μg/kg in all 7 silage samples tested; only traces (< 1 μg/kg) were found in only 2 of many samples from the rumen of cows. 3 of 24 processed cheeses flavoured with meat products and 1 containing herbs were NA-positive (10-73 and 20-24 μg/kg, resp.). Addition of $NaNO_2$ during processing resulted in the appearance of NA (1-2 μg/kg); this was prevented by ascorbate. [See FSTA (1978) 10 10P1408.] DMK

61

[Occurrence of nitrosamines in fodder, milk and milk products.] Untersuchungen zum Vorkommen von Nitrosaminen in Futtermitteln, Milch und Milchprodukten.

Terplan, G.; Hallermayer, E.; Kalbfus, W.; Unsinn, P.; Gärtner, C.-D.; Heerdegen, C.

Milchwissenschaft 33 (3) 142-145 (1978) [17 ref. De, en] [Lehrstuhl für Hygiene & Tech. der Milch, Univ., Munich, Federal Republic of Germany]

Samples to be examined were vacuum-distilled after comminution with water, NaCl and K_2CO_3, the distillate was extracted with methylene chloride, dried over anhydrous Na_2SO_4, concentrated and examined by MS on a Varian CH-5/GC 1440 outfit for presence of diethyl-, diisopropyl-, dipropyl- and dibutyl-nitrosamine, as well as of nitrosopyrrolidine and nitrosopiperidine. Diethylnitrosamine (DENA) was detected in 1 of 18 raw milk samples and in 1 of 20 heat-treated milk samples (at 2 μg/kg) but in none of 12 milk samples heated with nitrite. DENA was detected (at levels, when given, shown in parenthesis) in 2 of 12 samples of processed cheese, 3 of 8 processed cheese with ham (4-12 μg/kg), 1 of 5 processed cheese with salami (4 μg/kg), 1 of 10 other kinds of processed cheese (in which dipropylnitrosamine was also detected), 2 of 3 samples of processed cheese with added nitrite at > 50 p.p.m. (2-6 μg/kg), none of 10 samples of ham and cheese toast, 3 of 16 sauerkraut with salt meat (7 μg/kg; nitrosopyrrolidine was also detected at 3-8 μg/kg), none of 4 samples of sauerkraut with salt meat fermented with KNO_3 or $NaNO_2$, and in 1 sauerkraut with salt meat heated with added nitrite. Those samples containing DENA examined using a

Thermal Energy Analyzer were also found to contain small amounts of dimethylnitrosamine. SKK

62

Direct gas chromatographic analysis of cyclic N-nitrosamines.
Ray, S.
Journal of Chromatography 153 (1) 173-179 (1978) [27 ref. En] [Fisheries & Environment Canada, Bedford Inst. of Oceanography, Dartmouth, Nova Scotia B2Y 4A2, Canada]

A rapid gas chromatographic technique has been developed for quantitative estimation of cyclic N-nitrosamines. Cumbersome clean-up procedures are unnecessary and quantitative estimation can be done by injecting hexane extract without further pretreatment. The detection limit for the procedure is about 0.5 ng. Satisfactory results were obtained from analysis of fresh fish muscle spiked with 6 nitrosopiperidines at the 20 ng/kg level and extracted with hexane. Overall recoveries were 65-95%. Interfering peaks from the fish extract in the beginning of the chromatogram had no effect except on nitrosopyrrolidine. AL

63

[Separation and quantitative determination of volatile N-nitrosamines by high-performance liquid chromatography of DANS-derivatives.] Trennung und quantitative Bestimmung von flüchtigen Nitrosaminen durch Hochdruckflüssigkeitschromatographie von DANS-Derivaten.
Heyns, K.; Röper, H.; Stolzenburg, K.
Chemie Mikrobiologie Technologie der Lebensmittel 5 (5) 155-156 (1978) [12 ref. De, en, fr] [Inst. für Organische Chemie & Biochemie der Univ. Hamburg, Martin-Luther-King-Platz 6, D-2000 Hamburg 13, Federal Republic of Germany]

Suitable conditions for separation of DANS (1-dimethylaminonaphthalene-5-sulphonamide)-derivatives of low molecular dialkyl- and cycloalkylamines in a liquid chromatograph are described. A calibration curve for quantitative detn. of separated DANS-amides is given. The lower detection limit is 10 ng. AS

64

A study of the rates of the competitive nitrosations of pyrrolidine, p-cresol and L-cysteine hydrochloride.
Davies, R.; Massey, R. C.; McWeeny, D. J.
Journal of the Science of Food and Agriculture 29 (1) 62-70 (1978) [27 ref. En] [Min. of Agric., Fisheries & Food, Food Lab. (Colney), Colney Lane, Norwich NR4 7UA, UK]

The competitive nitrosations of pyrrolidine, p-cresol and L-cysteine hydrochloride were studied in aqueous solution. The reactions were studied at pH values 3.0, 5.0 and 5.5 and at temp. of 20°, 37° and 120° C, conditions which simulate those found in meat during processing, storage, cooking and gastric digestion. The formation of N-nitrosopyrrolidine was observed only in the reactions carried out at 120° C, subject to a detection limit of 0.2 p.p.m. At this temp. the effect of L-cysteine hydrochloride alone and in combination with p-cresol is to increase the rate of N-nitrosopyrrolidine formation at pH 5.0 but to inhibit formation at pH 3.0. In reactions at 20° and 37° C, where N-nitrosopyrrolidine was not detected, C-nitrosation of p-cresol to give o-nitro-p-cresol is inhibited in the presence of L-cysteine hydrochloride at pH values 3.0, 5.0 and 5.5. AS

65

Determination of N-nitroso compounds by mass spectrometry. A review. [Review]
Gough, T. A.
Analyst 103 (1229) 785-806 (1978) [200 ref. En] [Lab. of the Gov. Chem., Cornwall House, Stamford Street, London SE1 9NQ, UK]

Methods published in the literature for the detn. of N-nitroso compounds by MS, gas chromatography (GC)-MS, and practical GC-MS systems are considered, their relative merits are assessed, and the examination of foods (including bacon, other cured meat products, fish, cheese) and other compounds by GC-MS is reviewed. AL

66

Nitrates, nitrites and N-nitroso compounds. [Book]
World Health Organization; United Nations Environment Programme
Environmental Health Criteria No. 5, 107pp. ISBN 92-4-154065-6 (1978) [many ref. En] [Geneva, Switzerland]

This vol. is based primarily on original publications and reviews, references to which are contained in the 500-item bibliography. Following a summary of the major issues and recommendations for further studies, the book reviews the chemical properties of nitrates, nitrites and N-nitroso compounds and methods of their analysis; their sources in nature and elsewhere; their transport cycle and transformation in food and other substances; their environmental levels and exposure of the population; and their metabolism. Effects of nitrates, nitrites and N-nitroso compounds are considered in both animals and man, and health risks to man of exposure to these substances are evaluated. Available data indicate that the current concn. of nitrates and nitrites in food and water do not constitute a direct health risk for adults or older children, but there are risks, with possible fatal results, of high intakes of these substances in well water used for drinking or in making up infant foods and also in certain vegetables, for infants, particularly those <3 months of age. Attention is also drawn to the probable carcinogenic risk for man from exposure to N-nitroso compounds, most of which are mutagenic and some teratogenic in animal test systems. DMK

67

The fate of nitrite in meat curing and the determination of nitrosamines.
Woolford, G.
Dissertation Abstracts International, C 37 (1) 101 (1976) [En] [Univ. of Survey, Guildford, Survey, UK]

The metabolism of $NaNO_2$ by porcine skeletal muscle was investigated in vitro under conditions relevant to meat curing, i.e. at pH 6.0 under reducing (anaerobic) conditions with nitrite at 3mM (200 p.p.m.). Other concn. of nitrite were used to include the range relevant to curing and beyond. The rate of loss of nitrite from model cures was studied. In order to produce a 'balance sheet' the only stable N isotope, ^{15}N, was used as a tracer. Nitrite, labelled to 96.1% with ^{15}N, was found to be incorporated into both the non-water soluble fraction and the non-nitrite water-soluble fraction of the minced muscle cures. Analysis of the fractions revealed that enrichment was for the most part due to the formation of S-nitrosothiol derivatives and nitrosylmyoglobin. Analysis of the fractions also revealed the formation of nitrate and non-volatile N-nitrosamines, the latter in concn. ≤ 2 p.p.m. Total recovery of the ^{15}N label was 52-100%. Two methods of analysis of the ^{15}N were employed, MS and emission spectroscopy. Analysis of the headspace gases of the model cures by high resolution MS confirmed the formation of nitric oxide. Volatile N-nitrosamines were unequivocally identified by the development of 2 methods, both involving the use of high resolution MS and gas chromatography, in samples of foodstuffs preserved with nitrite prepared both commercially and in the laboratory. AS

68

Determination of volatile N-nitrosamines in bacon cook-out fat by nitrite release and thin-layer chromatography of fluorescent amine derivatives.
Cross, C. K.; Bharucha, K. R.; Telling, G. M.
Journal of Agricultural and Food Chemistry 26 (3) 657-660 (1978) [14 ref. En] [Canada Packers Ltd., Toronto, Ontario, M6N 1K4 Canada]

A method is described for the identification and detn. of volatile N-nitrosamines in bacon cook-out fat. The technique is based on separation by double distillation, clean-up on a column of silica gel, detn. by cleavage with HBr in glacial acetic acid, and subsequent colorimetric estimation of nitrite released and/or TLC of the 7-chloro-4-nitrobenzo-2-oxa-1,3-diazole derivatives of the corresponding amines. Lower limits of detection of N-nitrosodimethylamine and N-nitrosopyrrolidine (NPYR) are in the order of 5 μg/kg. The technique was compared with a high-resolution gas chromatography-MS method and gave comparable results. AS

69

Inhibition of formation of volatile nitrosamines in fried bacon by the use of cure-solubilized α-tocopherol.
Fiddler, W.; Pensabene, J. W.; Piotrowski, E. G.; Phillips, J. G.; Keating, J.; Mergens, W. J.; Newmark, H. L.
Journal of Agricultural and Food Chemistry 26 (3) 653-656 (1978) [28 ref. En] [E. Reg. Res. Center, USDA, Philadelphia, Pennsylvania 19118, USA]

Pork bellies were injected with a conventional cure formulation that would produce target levels of 125 p.p.m. $NaNO_2$ and either 500 p.p.m. sodium ascorbate or α-tocopherol alone or in combination. A mixture of α-tocopherol and Polysorbate 20 (1:0.4 w/w) dispersed in the cure produced a good distribution of α-tocopherol in the adipose tissue, which is thought to be the source of nitrosopyrrolidine precursor(s). The volatile nitrosamines in fried bacon were detected by GLC-thermal energy analyser and confirmed by GLC-high resolution MS. In a comparison of the same sections of each belly pair subjected to different treatments, a combination of α-tocopherol and sodium ascorbate or α-tocopherol alone was found to inhibit nitrosopyrrolidine formation more effectively than did ascorbate alone. AS

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